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Strain relaxation of epitaxial SiGe layers on Si(1 0 0) improved by hydrogen implantation

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Abstract

We propose a new method to fabricate strain relaxed high quality $Si_{1-x}Ge_x$ layers on Si by hydrogen implantation and thermal annealing. Hydrogen implantation is used to form a narrow defect band slightly below the SiGe/Si interface. During subsequent annealing hydrogen platelets and cavities form, giving rise to strongly enhanced strain relaxation in the SiGe epilayer. As compared to thermally induced strain relaxed Si–Ge epilayers, the hydrogen implanted and annealed samples show a greatly reduced threading dislocation density and a much higher degree of strain relaxation (90%). We assume that the hydrogen induced defect band promotes strain relaxation via preferred nucleation of dislocation loops in the defect band which extend to the interface to form misfit segments. The samples have been investigated by X-ray diffraction, Rutherford backscattering spectrometry and transmission electron microscopy.

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1. Introduction

The growth of high-quality $Si_{1-x}Ge_x$ (SiGe) epilayers on Si substrates is of great interest for both, fundamental research and device applications [1]. Due to the lattice mismatch between Si and Ge of 4.2%, strain is unavoidably introduced in epitaxial SiGe layers grown on Si. The strain will be either stored as strain energy in the film or accommodated by a network of misfit dislocations at the interface [2]. The first case can be realized only below a composition dependent critical thickness, where the in-plane lattice parameter of the SiGe layer adjusts perfectly to the Si substrate lattice. This growth mode – named pseudomorphic growth – implies a tetragonal distortion of the cubic SiGe lattice. In the second case, the epilayer thickness exceeds a critical value and the strain is greatly relieved by the generation of misfit dislocations at the SiGe/Si interface. Unfortunately, these misfit dislocations are accompanied by a high density of threading dislocations ($>10^9$ cm^{-2})

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which extend to the surface of the SiGe epilayer. Such high dislocation densities are unacceptable for electronic devices. As a rule of thumb, majority carrier devices may function with as many as 10^7 dislocations/cm^{-2}, whereas minority carrier devices require densities below 10^4 cm^{-2} [2]. On the other hand, the lattice mismatch strain at the SiGe/Si interface provides a means to adjust the valence and conduction band offsets, because strain modifies the electronic bandstructure. It can be accomplished not only by varying the Ge concentration in the SiGe layer but also by changing the strain conditions between the epitaxial overlayers and the substrate. Sufficiently large conduction band offsets for a Si quantum well – e.g. the essential part of a n-channel Si based quantum well device – requires a tensilely strained Si quantum well [1]. This can be achieved by growing the layer system on a so called virtual substrate, consisting of a strain relaxed SiGe buffer layer with a certain Ge concentration. Its in-plane lattice constant is determined by the Ge fraction and the degree of relaxation. The dislocation density and the interface roughness of such a buffer layer influences critically the electronic transport properties of the quantum wells. Best results have been obtained so far on several micron thick compositionally graded buffer layers [2,3]. However, both, the large thickness required and the remaining dislocation density of the order of 10^5 cm^{-2} are limiting applications.

Many different attempts have been made to solve this problem. Thin strain relaxed SiGe layers have been produced by implantation of Ge into thin Si layers on silicon on insulator (SOI) substrates [4]. Thermal annealing led to homogeneous SiGe layers directly on SiO_2, presumably promoted by the crystalline/amorphous SiGe/SiO_2 interface allowing strain relaxation by gliding. Alternatively, SiGe layers were grown on very thin silicon layers on SiO_2 (SOI wafers with nanometer thick Si layers) [5]. If the overgrown SiGe layer is thicker than the Si layer, the threading dislocations tend to bend towards the oxide interface preventing the formation of a high dislocation density in the overlayer structure. In another approach a Si buffer layer was grown at a low temperature (LT) by molecular beam epitaxy (MBE) [6–8]. The high defect density incorporated in the LT–Si buffer layer reduced the threading dislocation considerably. However, this approach led to promising results only for SiGe layers with Ge concentrations below ≈15 at.%. Recently, a new approach was proposed to solve the lattice mismatch problem and demonstrated for III/V materials. Wafer bonding was employed to form a twist boundary with an array of screw dislocations about 10 nm below the mismatched heterointerface [9]. The twist boundary seems to serve as a 'stretchable' atomic layer. The fabrication of such a compliant substrate by wafer bonding looks promising, however, the method is costly and technologically complicated.

In this study, we report an alternative method to reduce the threading dislocation density in relaxed SiGe epilayers by creating a defect layer in the substrate located close to the interface by hydrogen implantation.

2. Experimental

Two kinds of epitaxial $Si_{1-x}Ge_x$ layers with $x = 16.5$ (sample C1967) and $x = 22$ at.% Ge (sample B4081) were grown on Si(100) by solid source molecular beam epitaxy with thicknesses around 200 nm [1]. The 16.5% sample was fully strained as confirmed by X-ray diffraction, whereas the 22 at.% sample showed a slight relaxation of about 2% after growth. Hydrogen was implanted into the heterostructures with energies of ≈25 keV at room temperature and a dose of 3×10^{16} cm^{-2} with the samples tilted by an angle of 7°. After the implantation the samples were rapid thermally annealed (RTA) in Ar at 450°C for 30 s and 1100°C for 30 s. For comparison, similar samples were annealed without hydrogen implantation. Rutherford backscattering (RBS) was performed with 1.4 MeV He^+ ions at a scattering angle of 170°. The threading dislocation density was estimated from planar He ion channeling along {110} planes, which provides an improved sensitivity for threading dislocations as compared to axial channeling [10]. Cross-section (XTEM) and plane view transmission electron microscopy (TEM) were used to investigate the microstruc-

ture. The amount of strain relaxation was determined by high resolution X-ray diffraction.

3. Results

Fig. 1 shows the concept of our experiment. A hydrogen defected region was created slightly below (≈30 nm) the interface by hydrogen implantation through the SiGe/Si heterostructure. The width of the defect band was ≈90 nm. H-implantation mainly produces point-like defects [11]. Annealing around 400°C removed radiation damage within the near-surface region almost completely, whereas the end of range damage and the formation of hydrogen platelets were observed at 700°C. It is known that hydrogen stabilizes the defect band by strong Si–H-bonds. After annealing at 1000°C the defect band disappeared and cavities were produced in the range of the defect band. The implanted hydrogen doses were kept $\leqslant 3\times10^{16}\,\mathrm{cm}^{-2}$ to avoid blistering of the surface. At higher doses blistering occurs, an effect of the high pressure in hydrogen bubbles, where molecular hydrogen is formed due to the implantation and the subsequent annealing. The defect structure of the hydrogen introduced damage is described in the literature [11–13]. First experimental results are shown in the XTEM image (Fig. 2) of a $Si_{1-x}Ge_x$ sample with $x = 22$ at.% which was implanted with

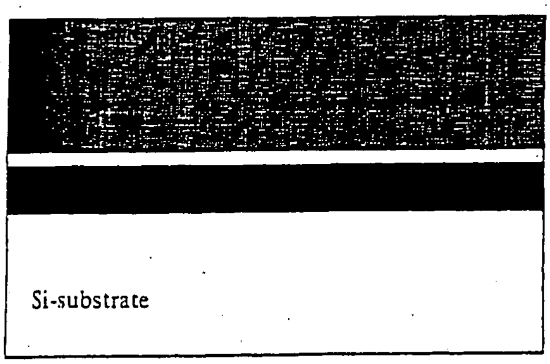

Fig. 1. Illustration of the concept of strain relaxation of a SiGe epilayer by hydrogen implantation. A defect layer is generated in close proximity to the lattice mismatch interface, which enhances strain relaxation by providing nucleation sites for misfit dislocations.

Fig. 2. XTEM-micrograph of a strain relaxed 22 at.% SiGe sample. The strain relaxation was obtained by hydrogen implantation and annealing. Strong strain contrasts are seen at the interface but no threading dislocations in the SiGe layer. The arrows mark hydrogen induced cavities which tend to interact with dislocations extending to the interface.

25 keV H^+ ions with a dose of $3\times10^{16}\,\mathrm{cm}^{-2}$ and annealed at 450°C for 30 s and 1100°C for 30 s. Most of the dark contrasts at the interface are due to strain. Surprisingly, no threading dislocations were observed in the SiGe-layer in the cross-section images. Instead, dislocations extend from the interface to the hydrogen cavities, which appear as circular contrasts. To enlighten the strain relaxation mechanism we present a random spectrum of the 22 at.% Ge sample and {1 1 0} planar aligned channeling spectra after different treatments in Fig. 3: after growth (pseudomorphic layer), after

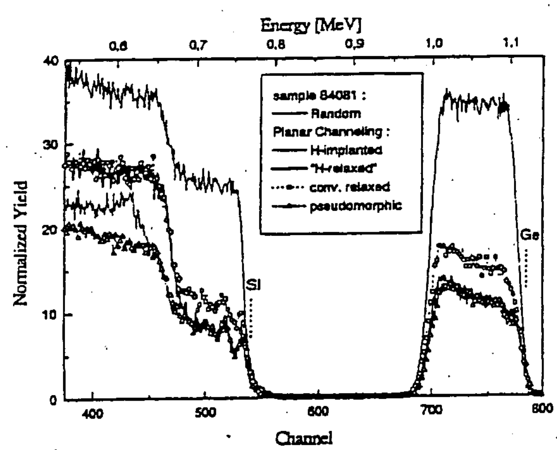

Fig. 3. Random and planar channeling of 1.4 MeV He^+ ions of a SiGe sample with 22 at.% Ge after various treatments as given in the inset. The hydrogen implanted and annealed sample (H-relaxed sample) shows the same dechanneling yield for the SiGe layer as the pseudomorphic layer, indicating a very low threading dislocation density.

hydrogen implantation (23 keV, 3×10^{16} cm^{-2}), after thermal annealing (conventionally relaxed) and after hydrogen implantation and annealing (H-relaxed sample). The planar dechanneling spectrum of the pseudomorphic layer serves as a reference for a (nearly) dislocation free sample. The near surface oscillations in the yields of the Si and Ge signals are fingerprints of planar channeling. The RBS-spectra of the as-implanted sample shows a dechanneling peak around channel number 430, where the end of range damage is placed, but no increased dechanneling in the Si–Ge layer. Annealing of the unimplanted sample at 1100°C for 30 s ('conventionally' relaxed) produced a strong increase in the backscattering yield in the epilayer and below the interface. The strong dechanneling at the interface arises from the evolution of a dense dislocation network, whereas the increase in the epilayer indicates the formation threading dislocations. Evaluating the dechanneling spectra provided a threading dislocation density of $N_{\mathrm{thread}} \approx 1.3\times10^{9}$ cm^{-2} [10]. In contrast, the H-relaxed sample shows no increase in the epilayer indicating a very low dislocation density. From the channeling results and the TEM cross-sections we estimated the density to be less than $N_{\mathrm{thread}} \leqslant 10^{7}$ cm^{-2}. A drastic increase in the backscattering yield is seen near the interface (channel number 470) indicating a dense misfit-dislocation network. Its total increase is larger than for the other samples indicating a higher misfit dislocation density at the interface. This is confirmed by the two plan-view TEM micrographs of Fig. 4 comparing the misfit dislocation networks in a thermally relaxed and a H-relaxed sample with a Ge concentration of 16.5 at.%. The difference is remarkable. The conventionally relaxed sample shows rather long misfit dislocation branches aligned in [1 1 0]-directions which are often piled up in bundles of several dislocations as typically observed in such buffer structures [14]. In comparison, the H-relaxed sample shows a much higher misfit dislocation density, patterned like a "patchwork" with a fairly regular mesh width. The misfit dislocations have a length in the range of only 40–100 nm. A higher misfit dislocation density suggests a higher degree of relaxation. X-ray diffraction results for the 16.5% sample are displayed in Fig. 5 showing the (400) reflections of the epi-

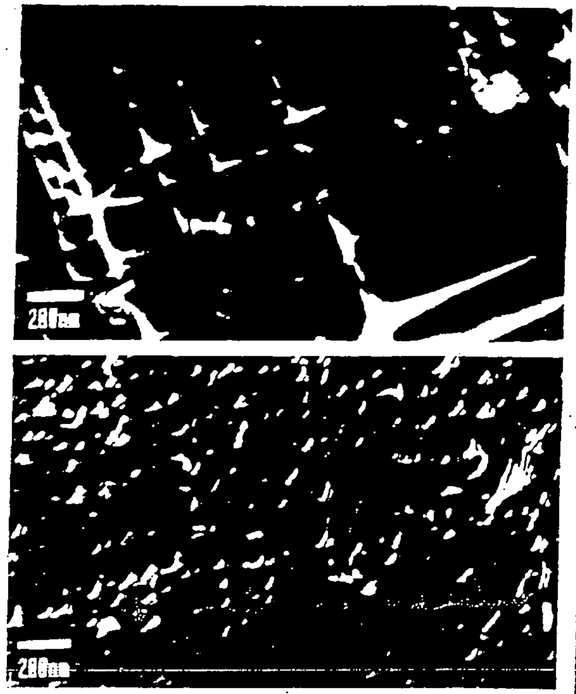

Fig. 4. Plan view TEM micrographs of differently strain relaxed SiGe samples with 16.5 at.% Ge. The upper micrograph shows the dislocation network at the interface after relaxation by RTA at 450°C and 1100°C for 30 s. The strongly curved line contrasts, more in the middle of the image, show threading dislocations. The lower image shows a very dense, but regular dislocation network achieved by hydrogen implantation and annealing.

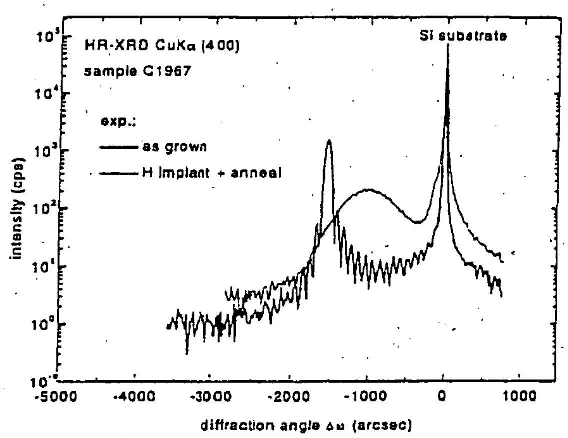

Fig. 5. X-ray diffraction of a SiGe sample with 16.5 at.% Ge before and after strain relaxation by hydrogen implantation and annealing.

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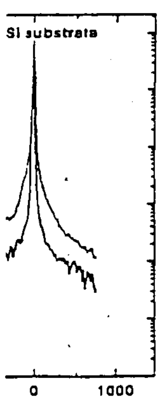

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layer and the substrate. The shift of the epilayer reflection towards the substrate signal provides the percentage of relaxation. About 90% relaxation was obtained for both H-relaxed samples, whereas only about 55% relaxation was measured for the conventionally relaxed samples receiving the same thermal treatment. As a further advantage of the H-relaxation, we found by atomic force microscopy that the surface roughness improved presumably because of the more regular misfit dislocation network (not shown).

How rough?

4. Discussion and conclusions

Introducing a defect band beneath the heterointerface greatly enhances the relaxation rate and drastically reduces the threading dislocation density in comparison to a conventionally relaxed sample. Follstaedt et al. found a strong interaction between dislocations and cavities formed by He implantation [15,16]. They could show that the introduction of a cavity layer right at the SiGe–Si interface strongly enhances the strain relaxation and, as a further benefit, that cavities efficiently getter metallic impurities. However, the number of threading dislocations in the epilayer could not be reduced notably in their experiments. Our attempt differs primarily in two ways: First, we used hydrogen instead of He and we placed the defect layer slightly below the heterointerface, although Follstaedt et al. [16] stated that cavities $\geqslant 20$ nm below the interface do not greatly perturb the misfit dislocation generation. It is known, that the evolution of defect structures after hydrogen implantation differs from that after He implantation [17]. Hydrogen forms primarily disk-shaped structures, so called hydrogen platelets [18]. These platelets form on various crystallographic planes. However, Aspar et al. observed that these platelets grow predominantly parallel to the (100) surface if a sufficiently thick cap layer – in our case a SiGe layer – is used [19]. This effect is used in the well known SMART CUT process, where hydrogen implantation and annealing is used to separate a thin silicon overlayer from the silicon substrate [19].

We assume that the defect band affects the strain relaxation during annealing in the following way. The defects promote nucleation of dislocation loops which may glide to the interface. There they act as misfit segments and enable strain relaxation. This assumption is also supported by the observation of the extremely dense dislocation network in the H-relaxed sample (Fig. 4). The strong interaction between dislocations and cavities is clearly seen in Fig. 2, where some dislocations end at the surface of cavities. The key point of this proposed mechanism is that strain relaxation of the SiGe layer may, in principle, occur without the formation of threading dislocations in the epilayer. Indeed, we could show a strong reduction of threading dislocation density in the SiGe layer in Figs. 2 and 3. However, we have to keep in mind that the conventional strain relaxation mechanism, nucleation and growth of misfit dislocations starting at the surface, may still occur as a competitive mechanism.

The observed relaxation process seems to have similarities with the mechanism observed for LT-MBE grown Si buffer layers, where in a defect rich buffer layer threading dislocations annihilate [7,8]. As mentioned above, this approach was so far only successful for smaller Ge concentrations. At higher Ge concentrations the conventional relaxation mechanism seems to dominate. There may be a concentration limit also for our approach, however, the very strong elastic interaction between hydrogen induced defects (primarily platelets and cavities) and dislocations may allow a more efficient confinement of threading dislocations below the epilayer. Since the elastic interaction decreases strongly with distance, the distance between interface and defect layer should play a crucial role. Further investigations are necessary to clarify this point. A further important observation is the improved surface smoothness of the H-relaxed sample showing only a very weak cross-hatch pattern. A plausible explanation is that the very dense, but regular dislocation network without dislocation bundles avoids the generation of surface undulations as usually observed for conventionally relaxed buffer layers [20].

In conclusion, hydrogen implantation enhances relaxation of strained Si–Ge layers on Si greatly. Strain relaxation preferentially occurs via nucleation and growth of dislocations primarily at the

end of range region of the implanted hydrogen. The H-relaxed Si–Ge layer adopts nearly its unstrained lattice parameter. The dislocation density in the Si–Ge layers is strongly reduced at least less than 10^7 cm^{-2}. Further experiments are required to investigate the dependence on the Ge concentration and the implantation and annealing conditions, as well as the precise threading dislocation density in the fairly thin SiGe-layer.

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